

PATENT SPECIFICATION



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COMPLETE SPECIFICATION.

A Process for Diminishing the Viscosity of Highly Viscous Cellulose Ethers.

We, Dr. ALEXANDER WACKER GESELLSCHAFT FÜR ELEKTROCHEMISCHE INDUSTRIE G.M.B.H., a body corporate organised according to the laws of Germany, of 20, Prinzregentenstrasse, Munich, Germany, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

In the manufacture of cellulose ethers most of the products obtained are not suitable for spinning and for making lacquers on account of their high viscosity.

Processes are known for diminishing the viscosity, but only at the expense of the mechanical properties of the product, since the ethers are more or less degraded. This deterioration is shown by the change in solubility, which is caused by an essential diminution of the micelle and a marked degradation. In fact, this effect has been used as a means of increasing the solubility of sparingly soluble or insoluble ethers, which for this purpose have been subjected to treatment with degrading or depolymerising agents: it is known that the solubility of cellulose ethers is increased, for instance, by treatment with mineral acids of suitable concentration, with or without the addition of a substance having a solvent or swelling action on the ether, or by treatment with glacial acetic acid in presence of a small proportion of sulphuric acid.

In the process of the present invention the mechanical properties of the product remain satisfactory although a profound diminution of viscosity is attained. Degradation is completely avoided, as may be recognised by the unchanged solubility of the material.

According to the invention, the viscosity of cellulose ethers is diminished without varying the solubility of the ethers, by treating the ethers with a hot aqueous mineral acid of relatively low concentration (that is to say, a mineral acid of concentration lower than that at which the acid is known to have a degrading or depolymerising action on the ether, with its attendant alteration in the solubility of the ether), or a hot solution of an acid salt in water, with exclusion of swelling agents and solvents. Similar results are also obtained according to the invention by treating the cellulose ethers for some time with hot aqueous acetic acid of suitable concentration, without the use of hydrolytically acting agents, such as mineral acids or the like. The duration of the heating depends both on the concentration of the acid and on the temperature.

By either treatment the viscosity of the treated material is essentially lower than that of the original material, so that the application of the treated material for making varnishes and for spinning becomes possible. The mechanical properties, like tenacity and elasticity are not unfavourably influenced by the treatment, since as is shown by the unchanged solubility, no degradation has occurred.

The following Examples illustrate the invention, the parts being by weight and the figures representing the viscosities being relative and referring in all cases to solutions of the ethers in a mixture of dichlorethylene and alcohol: in each Example, the ethyl cellulose used as parent material has a content of ethoxyl of 40 per cent.

EXAMPLE 1.

1 part of ethylcellulose, having a primary viscosity of 800, is heated for $\frac{1}{2}$ to 6 hours at about 95° C. with 5—20 parts of acetic acid of 80 per cent. strength.

The diminution in viscosity is shown by the following figures:—

Viscosity after 15 minutes treatment:	445
„ „ 5 hours	62
„ „ 6 „	50

The solution, hot or cold, and, if necessary, after filtration, is mixed with water to form a precipitate which is washed and dried.

EXAMPLE 2.

1 part of ethylcellulose, having a primary viscosity of 150 is suspended in 5—20 parts of acetic acid of 30 per cent. strength and the suspension is heated for 3 hours, preferably while stirring, at the boiling point. After this time the vis-

cosity is diminished to 115. The material is then washed with water and dried.

EXAMPLE 3.

1 part of ethylcellulose, having a primary viscosity of 665, is heated for an hour with 10—30 parts of a solution, containing 2 per cent. of hydrochloric acid. After the treatment the viscosity has diminished to 116; the material is then washed and dried.

EXAMPLE 4.

1 part of ethylcellulose, having a primary viscosity of 665, is heated for 3 hours with 5—20 parts of a solution of sodium bisulphate of 2 per cent. strength. After the treatment the viscosity has diminished to 316.

The heating operations may be conducted at the ordinary pressure or in an autoclave under elevated pressure.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. A process for diminishing the viscosity of cellulose ethers without changing their solubility, wherein the cellulose ethers are treated with a hot aqueous mineral acid of relatively low concentration or with an aqueous solution of an acid salt, with exclusion of swelling agents and solvents. 30

2. A process for diminishing the viscosity of cellulose ethers without changing their solubility, wherein the cellulose ethers are heated with dilute or concentrated aqueous acetic acid without addition of a hydrolysing agent or a depolymerising or degrading agent. 35 40

3. Cellulose ethers of diminished viscosity which can be made by the process referred to in claim 1 or 2, whenever so made or made by any process which is the obvious chemical equivalent thereof. 45

Dated this 8th day of November, 1929.

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